

# Inorganic Membrane Porous Support Tube Fabrication

Brian Bischoff and Roddie Judkins

Oak Ridge National Laboratory

Presented at

DOE Hydrogen Program Annual Review  
Crystal City, VA

May 25, 2005  
Project #PDP18

This presentation does not contain any proprietary or confidential information

\*Oak Ridge National Laboratory is managed by UT-Battelle, LLC, for the U.S. Department of Energy under Contract No. DE-AC05-00OR22725. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes.

# Project Objectives

- To develop porous metal supports for hydrogen separation membranes that are compatible with the supported membrane and operational environment

# Budget

	Budget (k\$)
FY2004	100
FY2005	0*

\*The budget request was \$200K. However, due to changes in EERE priorities the funding zeroed out for FY05.

# Technical Targets

## ➤ DOE Technical Barriers

- A. Fuel Processor Capital Costs
- B. Operation and Maintenance Costs
- AB. Hydrogen Separation and Purification

## ➤ DOE Technical Targets for 2010

- Purification: 90% at \$0.03/kg Hydrogen
- Palladium Membranes: <\$100/ft<sup>2</sup> capable of operating at 300-600 °C for 100,000 hrs with at flux of 200 scfh/ft<sup>2</sup>

# Technical Approach

Develop a composite support tube structure especially for palladium membranes

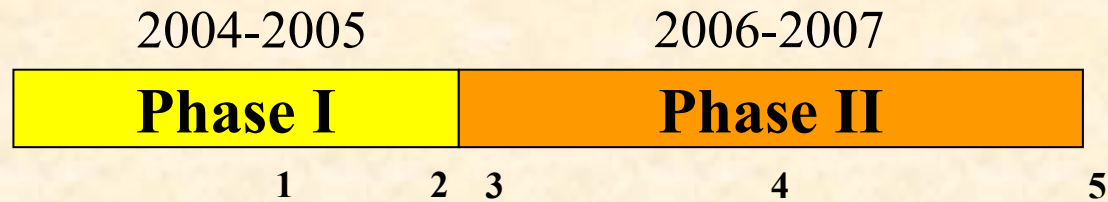
## ➤ Approach for Porous Support Tube Development

- Establish performance criteria for support tubes for palladium, microporous, ion-transport membranes
- Identify potential support tube materials and down select through a rigorous investigation of potential for fabrication and compatibility with Pd (initially)
- Establish fabrication protocols

# Project Timeline

(Project initiated February 2004)

## Support Tube Development



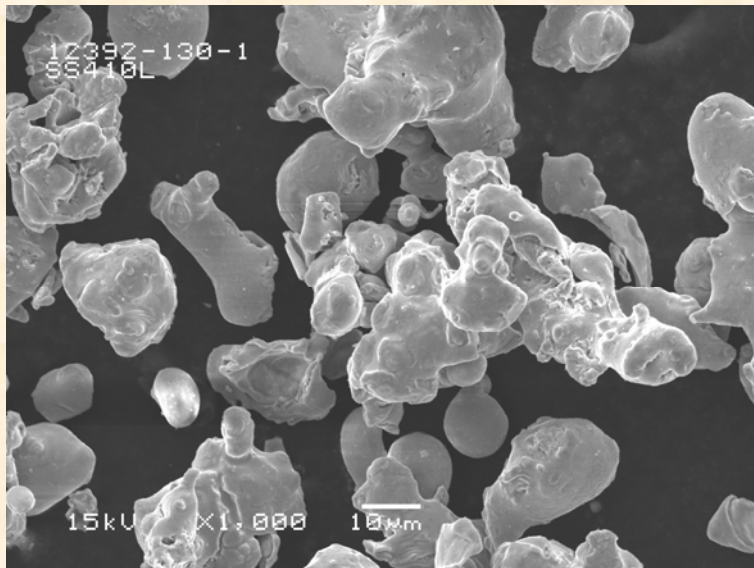
- Phase I: Development and Testing
  - 1 – **Prototype Support Tube**
  - 2 – Complete tests to determine efficacy of tubes to accommodate membrane layer(s)
- Phase II: Optimization, Scale up and Tech Transfer
  - 3 – Composite Support Development (initiate)
  - 4 – Complete tests to determine efficacy of composite tubes to accommodate membrane layer(s)
  - 5 – Technology Transfer

# Technical Progress

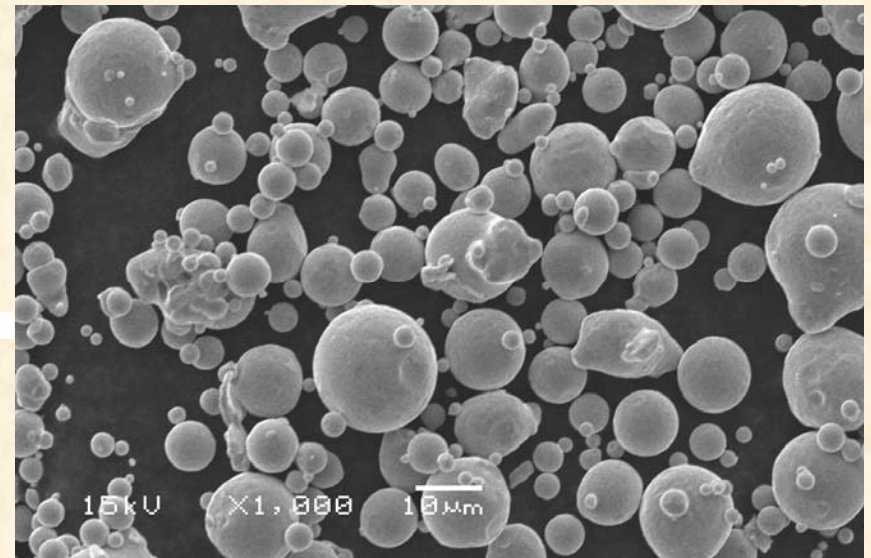
## (Porous Support Tube Development)

- Potential support tube materials have been identified and include:
  - 300 and 400 series stainless steels,
  - Iron Aluminide, and
  - Hastelloy X
- Gas (argon or helium) atomized powders have greatest potential for hydrogen membrane supports (powders are spherical and size distribution can be controlled)
- Support tube forming process parameters are being established
- Palladium membranes need barrier layer to prevent intermetallic diffusion of metal atoms from support into palladium membrane layer. Future work will include application of this intermediate layer.

# Gas Atomized Powder is More Spherical



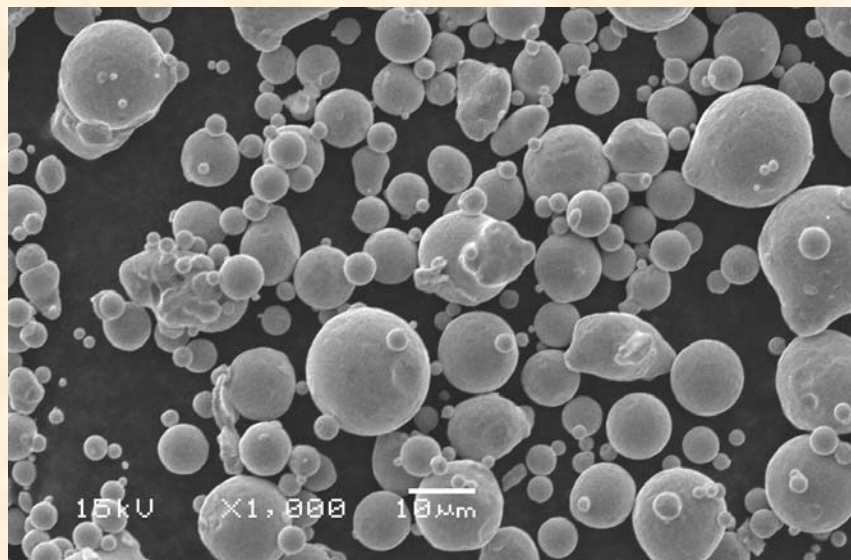
Water Atomized 410 Stainless Steel  
Powder



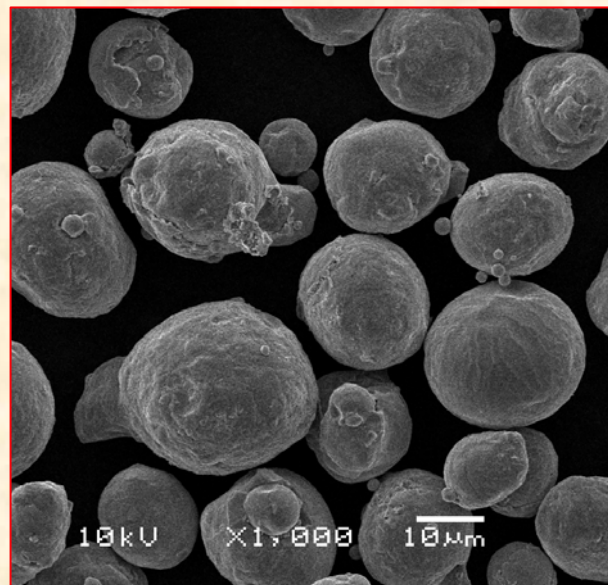
Gas Atomized 410 Stainless Steel  
Powder



# Uniform Particle Size is Key to High Quality Supports



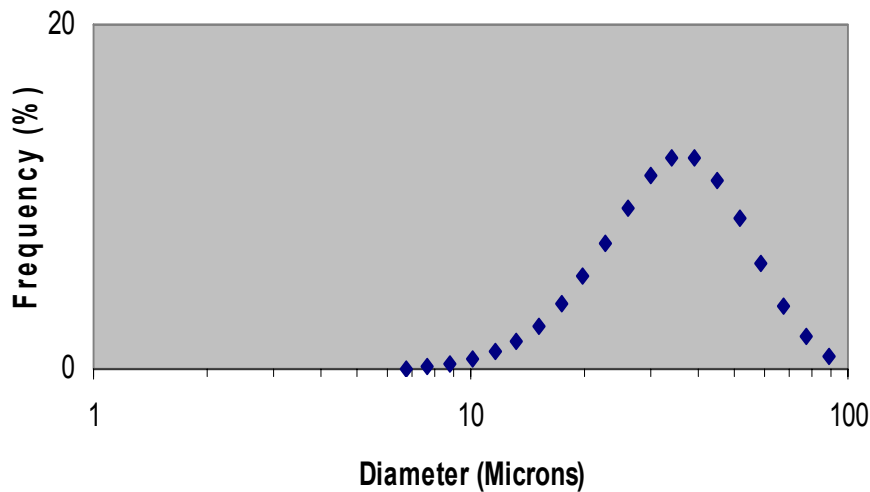
Standard Gas Atomization Produces  
Broad Size Range of Powder



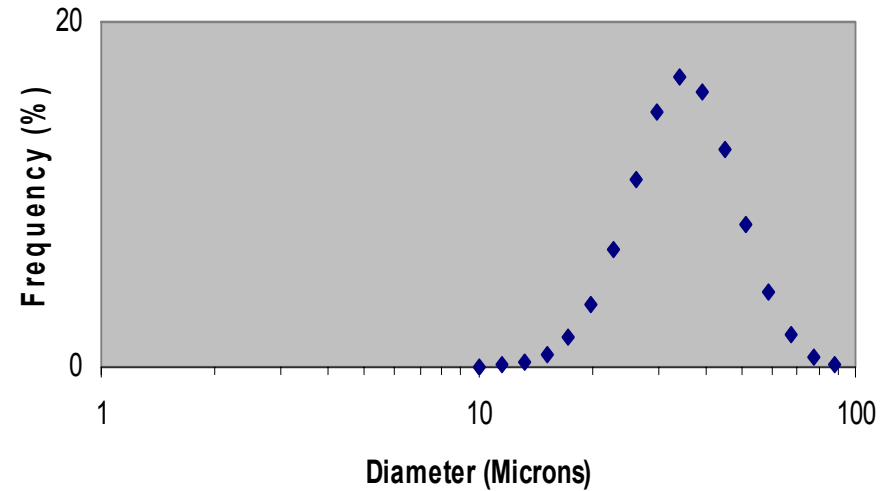
Gas Atomization at Ames  
Laboratory Produces Uniform  
Particle Size

# Ames Laboratory Can Fabricate Spherical Particles With a Very Narrow Size Distribution

Particle Size Distribution of Water Atomized Powder



Particle Size Distribution of Gas Atomized Powder from Ames



# Interactions and Collaborations

- **Ames Laboratory:** providing novel materials for support tubes
- **Worcester Polytechnic Institute:** discussions were planned to have WPI deposit Pd membranes on ORNL support tubes
- **NETL:** initial discussions on collaborative effort
- Discussions on implementation of technology are ongoing with
  - **ConocoPhillips, ChevronTexaco, Pall Corp., and Praxair**

# Future Work

- Porous Support Tube Development
  - Continue to identify and characterize materials for support tube fabrication
  - Establish fabrication parameters and fabricate support tubes FY2005
  - Characterize support tubes for strength, permeance, and high temperature stability
  - Expand activity to include composite structure support tubes

# Project Safety

*The most significant hazard is the use of pure hydrogen in our membrane test systems*

*Our approach to ensuring safe operation includes:*

- Project has undergone “Integrated Safety Management Pre-Planning and Work Control” (Research Hazard Analysis and Control)
- Each work process is authorized on the basis of a Research Safety Summary (RSS) reviewed by ESH subject matter experts and approved by PI’s and cognizant managers
- The RSS is reviewed/revised yearly, or sooner if a change in the work results in a need for modification.
- Experienced Subject Matter Experts are required for all Work Control for Hydrogen R&D including periodic safety reviews of installed systems
- Results of Work Control Process requires:
  - Monitoring hydrogen concentration at ceiling above test system. Alarm sounds at 50% LEL.
  - Personnel be present at all times when using hydrogen.
  - Evacuation of gas lines of air or purging with inert gas prior to introduction of hydrogen
  - Exhaust of gas lines containing hydrogen using eductors instead of electrically driven vacuum pumps.